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Cementation and Solidification of Contaminated CERCLA Wastes from the 903 Pad/OU2

SUMMARY TEST PLAN
FOR CEMENT ENCAPSULATION OF CONTAMINATED SOILS

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ENVIRONMENTAL REMEDIATION SOLIDIFICATION DEVELOPMENT - MISCELLANEOUS WASTE TREATMENT

SUMMARY TEST PLAN FOR CEMENT ENCAPSULATION OF SURROGATE AND RADIOACTIVELY CONTAMINATED SOILS

1 0 INTRODUCTION

The Rocky Flats Environmental Technology Site (RFETS) has several locations that are contaminated with organic compounds and various radionuclides The soil may also contain various Resource Conservation and Recovery Act (RCRA) listed toxic metals (e g cadmium and chromium) The treated waste must be RCRA land disposal restricted (LDR) compliant as determined by the Toxicity Characteristic Leaching Procedure (TCLP) tests prior to disposal Cementation is a viable solidification process for a variety of the mixed radioactive waste forms at RFETS and is an ideal technology for treating both aqueous and solid waste to bring them into compliance with LDR requirements

Primary development efforts for cementation technologies have focused on the treatment of radioactive nitrate salts created by the evaporation of the plant s aqueous waste in Building 374 Additionally analytical laboratory waste solutions have been treated using the Bottle Box cementation process to immobilize RCRA listed heavy metals in this mixed waste stream

2 0 OBJECTIVES

Demonstration of cementation treatment of contaminated soil wastes will involve two basic activities a cold test with surrogate materials followed by a hot test using real waste This test plan addresses both tasks in a summary fashion with the objective to demonstrate encapsulation of a contaminated soil waste and to optimize the cementation formulation This will be accomplished in cold testing using non radioactively contaminated materials as surrogates TCLP testing will also be performed on the cemented surrogate soil residue to verify that the treated soil will be LDR compliant The surrogate soil will be selected to be as consistent as possible with actual wastes based on available waste characterization data Laboratory facilities located in Building 881 will be used for the cold test Once the formulation is optimized based on information obtained from these tests actual wastes will be cemented facilities in Building 779 will be used for the hot work with actual soil samples Samples of the cemented waste will also have to be evaluated to confirm the cemented waste is LDR compliant

3 0 BACKGROUND

The 903 Pad area was used as an underground drum storage site from 1958 until 1967 These drums contained radioactively contaminated oils (mineral oils carbon tetrachloride hydraulic oils pump oil silicone oils etc) with most of the waste oil stored in 55-gallon drums Over time an estimated 5 000 gallons of liquid contaminated with plutonium leaked from the drums into the soil Several treatment technologies being considered for this soil may leave a soil residue containing high concentrations of heavy metals and/or radionuclides which require further treatment prior to disposal Cementation is one technology being considered for making this residue LDR compliant

4 0 SAFETY AND REGULATORY ISSUES

4 1 Safety Analysis

Only general industrial hazards will be associated with this cold demonstration No additional special hazards are anticipated

4 2 National Environmental Policy Act (NEPA)

A Memo to File was approved in 1990 for experiments using surrogate wastes A Categorical Exclusion (CX) was approved in 1992 for experiments using wastes

4 3 Resource Conservation and Recovery Act (RCRA)

A Treatability Study Exemption (TSE) will be required for experiments using actual wastes

4 4 Clean Air Act (CAA)

No materials listed as pollutants in the CAA will be involved in these cold tests

5 0 APPROACH

Cementation development will take place in two phases Phase I will evaluate the feasibility of the cementation process for the surrogate contaminated soil while Phase II will involve the cementation and stabilization of an actual soil residue from the 903 Pad After the cement formulation is optimized with respect to waste loading etc a surrogate soil residue will be spiked with RCRA listed heavy metals (i e Cd Ba Cr) at "worst case levels plus 10 / and cemented These samples will be submitted for TCLP to confirm that the stabilized soil residue is LDR compliant All cold experiments will take place in Building 881 Rooms 267 and 296 using an existing Hobart mixer to prepare test samples The experiments will be conducted using a surrogate soil residue based on available waste characterization data

A Taguchi experiment has been designed to evaluate fly ash addition (variable A) waste loading level (variable B) water/cement ratio (variable C) and cement type (variable D) Additionally interactions between variables A and B A and C and B and C will also be evaluated This will require eight experimental runs to complete the study (refer to Table 1) Data collected during the series of experiments will include waste form density waste volume changes viscosity and freeze/thaw performance of the product

Once the cement formulation is optimized with respect to waste loading etc samples of actual soil from the 903 Pad/OU2 will be cemented These samples will also require verification testing to insure that the waste form is LDR compliant (i e passes TCLP testing for RCRA metals)

6 0 WASTE ACCEPTANCE CRITERIA

The final stabilized product must meet the disposal site waste acceptance criteria (WAC) including Department of Transportation (DOT) and RCRA regulations and regulatory requirements for LDR wastes The WAC includes but is not limited to the following

The residues will require immobilization if
1 wt/ of the soil has a particle diameter of < 10 µ or
15 wt/ of the soil has a particle diameter of < 200 µ

The soil must not contain free liquids

Soil residues shall be treated to reduce volume and provide a more physically and chemically stable waste form where practical

Residues shall not react significantly with the packaging during normal storage shipping and handling and

Mixed wastes prohibited from land disposal under 40 CFR § 268 will not be accepted unless treated as specified under 40 CFR § 268 Subpart D

A visual inspection for the presence of free liquids and dispersible fines using a PASS FAIL criteria will be used in this preliminary testing Any samples showing the presence of free liquids or dispersible fines will be rejected

The physical stability of the waste form will be evaluated through freeze/thaw cycling experiments This will measure the weight and dimensional changes over multiple thermal cycles to determine whether the waste form is suitable to long term storage The viscosity will be measured as a function of waste loading for each formulation This will determine whether the cement waste mixture is amenable to mixing in standard equipment Waste form density will provide a measure for the volume changes that can be expected with cementing this waste stream and is accomplished by comparing the soil volume with the cemented volume

7 0 QUALITY ASSURANCE

7 1 Planning

This summary test plan is an upper level outline of the cold testing effort In addition to this document each individual experiment will be controlled by an experimental plan Experimental plans provide more detail on specific objectives and test procedures for the particular experiment The experimental plans will be implemented using run sheets on which the raw experimental data are recorded

7 2 Data Collection and Analysis

The cold testing effort is basically a feasibility investigation designed to demonstrate the cementation of contaminated soil waste Therefore at least initially a rigorous quality approach is not planned Only minimal equipment will be used to collect data The majority of the data to be collected during this effort will be qualitative for example the flow characteristics of the cement and a visual evaluation of how well the cement encapsulates the soil

7 3 Reporting

A final report is required at the end of both the cold testing and hot testing portions of this effort The final report will document the test results provide an analysis of the results and suggest any follow up testing These reports will go through a peer review within the development team as well as a review by Technology Development management

All test plans experimental plans run sheets and reports will become a record of this effort All records will be maintained in the project files

8 0 REFERENCES

1 U S Department of Energy June 1992 Comprehensive Treatment and Management Plan Version 1 3 Rocky Flats Office Golden Colorado

2 Work Plan for Chemically Enhanced Steam Stripping of Radionuclides in RFP Soils Environmental Restoration Program RFP/ER 94 00003 Rev 0 EG&G Rocky Flats Inc Rocky Flats Plant April 1994

Table 1 Taguchi L_8 Designed Experimental Matrix for Cement Stabilization of Surrogate OU2 Soil Residues

Trial	Fly Ash Loading (A)	Waste Loading (B)	(AxB)	Water/Cement (C)	(AxC)	(BxC)	Cement Type (D)
1	0 /	20 /	1	0 55	1	1	I/II
2	0 /	20 /	1	0 70	2	2	V
3	0 /	35 /	2	0 55	1	2	V
4	0 /	35 /	2	0 70	2	1	I/II
5	≈23 %	20 /	2	0 55	2	1	V
6	≈23 %	20 /	2	0 70	1	2	I/II
7	≈23 /	35 /	1	0 55	2	2	I/II
8	≈23 %	35 /	1	0 70	1	1	V

ENVIRONMENTAL REMEDIATION
SOLIDIFICATION DEVELOPMENT

EXPERIMENTAL TEST PLAN OU2 001

CEMENT ENCAPSULATION OF SURROGATE OU2 CONTAMINATED SOIL RESIDUE

OBJECTIVE

The objective of this experiment is to optimize the cement formulation for the encapsulation of the OU2 contaminated soils such that the final form meets Resource Conservation and Recovery Act (RCRA) requirements

A surrogate soil residue will be used in these tests to represent the actual residues from the OU2 Site/903 Pad

TEST PROCEDURE

1 0 Weigh out the surrogate soil residue cement and water

- 1 1 Select the type of cement (type I/II or V) required for the experiment
- 1 2 Weigh out the quantity of cement to be used in the experiment
- 1 3 Weigh out the quantity of class F fly ash if required
- 1 4 Weigh out the proper amount of surrogate contaminated soil and record the amount added (soil quantity will be added as wt / of powder and includes water cement and fly ash) Measure and record the approximate bulk density of the soil
- 1 5 Weigh out the quantity of water required

2 0 Measure weight of plastic vials

- 2 1 Obtain eight plastic vials of 160 ml capacity
- 2 2 Label plastic vials for identification purposes with a permanent marker (Example 1 3 for experiment #1 sample #3)
- 2 2 Measure and record the weight of the empty vials in grams to the nearest tenth of a gram

3 0 Mix the soil and cement with the water

- 3 1 Pour the water into the Hobart container
- 3 2 Pour the soil into the container and mix manually with a spatula for up to 1 minute
- 3 3 Pour the cement and fly ash (if required) into the slurry and mix manually with a spatula for up to 1 minute
- 3 4 Turn Hobart mixer on at low speed setting (setting #1) and mix for 30 seconds
- 3 5 After 30 seconds turn mixer off and scrape sides of container using spatula
- 3 6 Turn Hobart mixer on at the low setting and mix for an additional 1 minute
- 3 7 Record the approximate final volume of the mixture

4 0 Measure the viscosity of the cement mixture

4 1 Place sample of cement into viscometer stainless steel canister

4 2 Position canister in EG&G Chandler Model 35 viscometer and wait two (2) minutes

4 3 Turn viscometer on at highest rpm setting (600) Measure the degree of deflection after five seconds and record value

4 4 Reduce rpm setting to next lowest setting (300) Measure the degree of deflection after five seconds and record value

4 5 Continue until readings have been measured on all settings

4 6 Clean viscometer

5 0 Measure the packed bulk density of the cement

5 1 Carefully pack cement into plastic vials Avoid getting any cement on the outer sides of the vials

5 2 Tap the vials repeatedly on a table to remove any voids in the cement

5 3 When the vials have been filled measure the weight of the filled vial to the nearest tenth of a gram

5 4 *Subtract the empty vial weight (Step 3 2) from the filled vial weight*

5 5 Calculate and record the waste form density (WFD = [Filled Weight Empty Weight]/ 169 [actual vial volume in cubic centimeters])

5 6 Discard the remainder of the cement (plastic bucket or bag) and clean the Hobart mixer and area Place wastes in proper receptacle for non hazardous wastes

6 0 Cure the cement

6 1 *Place all but one of the vials in an oven heated to 50 ± 5 C and cure for 24-48* hours

6 2 Remove the cured samples from the oven

6 3 Remove the cement from the plastic and record the sample number on cement using a permanent marker

7 0 Perform Freeze/Thaw Study

7 1 Measure and record weight on samples 1 through 3 from each formulation

7 2 Place samples in freezer and cool to 25 ± 5 C overnight

7 3 Remove samples and thaw in drying oven set to 50 ± 5 C

7 4 Measure and record weight of the samples along with appropriate observations

7 5 Complete for a total of three freeze/thaw cycle tests for sample #1 Complete for a total of six cycles for sample #2 and 12 cycles for sample #3

7 6 Photograph the samples

Test Plan Originator

Team Lead

ENVIRONMENTAL REMEDIATION SOLIDIFICATION DEVELOPMENT

EXPERIMENTAL TEST PLAN OU2 002

TCLP DEMONSTRATION OF CEMENTED SURROGATE OU2 CONTAMINATED SOIL RESIDUE

OBJECTIVE

The objective of this experiment is to demonstrate that a cemented surrogate soil residue spiked with Resource Conservation and Recovery Act (RCRA) listed metals passes the Toxicity Characteristic Leaching Procedure (TCLP) in order to be Land Disposal Restricted (LDR) compliant The RCRA constituents will be added at a pre determined level based on available characterization data the amount of RCRA metals added to the soil residue will be "worst case plus 10%

A surrogate soil residue will be used in these tests to represent the actual residues from the OU2 Site/903 Pad

TEST PROCEDURE

1 0 Weigh out the surrogate soil residue RCRA constituents cement and water

- 1 1 Weigh out and record the individual quantities of the RCRA heavy metal constituents to the nearest one thousandths of a gram The heavy metals will added in the form of nitrates hydrated nitrates or hydrated sulfates
- 1 2 Weigh out and record the quantity of cement to be used in the experiment
- 1 3 Weigh out and record the quantity of class F fly ash if required
- 1 4 Weigh out and record the proper amount of surrogate contaminated soil and record the amount added (soil quantity will be added as wt% of powder and includes water cement and fly ash) Measure and record the approximate bulk density of the soil
- 1 5 Weigh out and record the quantity of water required

2 0 Measure weight of plastic beakers

- 2 1 Obtain five plastic beakers of 400 ml capacity
- 2 2 Label plastic vials for identification purposes with a permanent marker
- 2 2 Measure and record the weight of the empty beakers in grams to the nearest tenth of a gram

3 0 Mix the soil and cement with the water

- 3 1 Pour the water into the Hobart container
- 3 2 Add the soil and the RCRA additives to the water and mix manually with a spatula for up to 1 minute
- 3 3 Pour the cement and fly ash (if required) into the slurry and mix manually with a spatula for up to 1 minute

3 4 Turn Hobart mixer on at low speed setting (setting #1) and mix for 30 seconds
3 5 After 30 seconds turn mixer off and scrape sides of container using spatula
3 6 Turn Hobart mixer on at the low setting and mix for an additional 1 minute
3 7 Record the approximate final volume of the mixture

4 0 Measure the packed bulk density of the cement

4 1 Carefully pack cement into plastic beakers placing a uniform amount into each beaker Avoid getting any cement on the outer sides of the vials
4 2 Tap the beakers repeatedly on a table to remove any voids in the cement
4 3 When the beakers have been filled measure the weight of the filled beaker to the nearest tenth of a gram Measure the filled volume to the nearest 10 ml
4 4 Subtract the empty beaker weight (Step 3 2) from the filled vial weight
4 5 Calculate and record the waste form density (WFD = [Filled Weight Empty Weight]/ Material volume
4 6 Clean equipment and area Place any wastes in proper receptacle for hazardous wastes

5 0 Cure the cement and perform TCLP analysis

5 1 Place the beakers in an oven heated to 50 ± 5 C and cure for 24 48 hours
5 2 Remove the cured samples from the oven
5 3 Transfer the samples to Environmental Technology for TCLP testing

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Test Plan Originator Team Lead